LABORATORY EQUIPMENT AND SUPPLIES FOR SEWAGE TESTS

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Bachelor of Science

Oklahoma Agricultural and Mechanical College

1949

Submitted to the School of Civil Engineering

Oklahoma Institute of Technology of the

Oklahoma Agricultural and Mechanical College

for the Degree of

MASTER OF SCIENCE

1950

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PREFACE

The primary objective of this report is to discuss and recommend certain basic tests that may be used at the small sewage treatment-plant laboratory. It also attempts to determine the cost of supplies and instruments to equip a laboratory.

It also discusses tests to be performed at the larger plants with better equipped laboratories.

The tables list the number of pieces of equipment used in the various tests and the present unit and total cost to make the recommended examinations.

The writer respectfully acknowledges his indebtedness to Professor Quintin B. Graves and Nathan C. Burbank of Oklahoma A. and M. College, for their invaluable assistance in the preparation of this report. He also wishes to acknowledge the assistance rendered by C. S. Hays of the Stillwater sewage treatment-plant for his discussions and demonstrations in the various phases of plant operation, and to thank the authors of all material used.

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January, 1950

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A very important factor in the design of a sewage-treatment plant is the determination of the substances existing in the sewage that may influence the type of treatment required and effect the degree of treatment necessary to meet the demands for the safeguarding of public health. Equally important, and very closely related, are the tests that furnish information on which the operation of the plant may be based. The efficiency of the plant may also be determined by making certain laboratory tests.

Since the success in operation of the sewage-treatment depends in a large part on the test results, every plant, regardless of size or type, should be equipped with a testing laboratory. The tests made in the laboratory are determined by the type of the plant, the character of the wastes, the amount of diluting water, degree of treatment and related factors.

The composition of sewage from different cities varies widely. This is affected some by type of industries that may exist. However, there may be a wide variation in the quality of sewage in a given city. During the night the sewage is usually weak, while during the day it may be strong. It may also vary with the day of the week. For examination, a large number of samples must be taken at different times and the results interpreted accordingly.

The amount of diluting water may also vary from day to day but the effluent put into streams should be such that on days with least diluting flow, it will not endanger fish life nor create a nuisance.

Sewage treatment plants may vary in size and type with different cities. The treatment may vary slightly in different plants even though the same process is employed. The laboratory tests and equipment for the following types will be discussed: (1) plain sedimentation, (2) chemical precipitation and (3) activated sludge process.

Sampling the sewage for the various types of plants is somewhat the same. Care should be taken to acquire a sample that best represents the entire flow.

Sewage is an ever-changing mixture of substances in solution and in suspension. Changes take place both with time and with place. To secure a representative sample is difficult.

There is no ideal time to sample sewage as its quality is varying continuously. A sample may be taken at any moment but its analysis must be interpreted only on the basis of the conditions at that particular moment. A sample taken earlier or later would probably show different results.

The general character of sewage is best ascertained by mixing samples taken every hour or half hour for 24 hours and analyzing the composite sample. For very accurate determinations, the quantities of half-hourly samples mixed together should be proportionate to the flow of sewage at the time of sampling. As some of the suspended matter may be quite coarse, it is desirable to take 1 quart or more of sewage at each sampling and pour it into a clean basin or barrel. The contents of the barrel should be thoroughly mixed at the end of 24 hours and from 2 to 4 quarts taken from the composite for analysis. When small samples are taken for laboratory tests, care should be exercised to make sure it is fairly representative of the large sample.

Because the quality of sewage may change during the hours that the composite sample is being made up, it is necessary to keep the samples cool. The sample should be kept at a temperature between 4 and 10 degrees C., or some form of preservative may be placed in the collecting basin to inhibit the biologic action that otherwise would occur. Suitable preservatives include chloroform, formaldehyde, and sulphuric acid. A preservative must be selected that will not affect the results of the analyses to be made. For example, no preservative can be used in a sample on which determinations of biochemical oxygen demand are to be made. Determination of this important characteristic cannot be made successfully on 24-hour composite samples of a strong sewage.

The procedure to follow in sampling wet sludge will depend upon where the sample is collected. If the sample is drawn from a pipe line through which the material is flowing, a small quantity should be collected at regular intervals and placed in a wide mouthed bottle or bucket. If there is a valve for withdrawing the sample, this valve should always be left open long enough to free the pipe of sludge between the valve and the main pipe line before collecting the sample. Samples collected at different depths in Imhoff and separate sludge-digestion tanks are somewhat more difficult to obtain. One method is to provide a light pole a few feet longer than the depth of the tank. Attached to the bottom of the pole should be a wide mouthed bottle. The bottle should contain a stopper attached to a string. By lowering the bottle to the desired depth and removing the stopper by use of the string, the sample can be taken. Another method is to use a pitcher pump with a varable length suction pipe. The suction end is lowered to the level where the sample is to be taken and the sludge is pumped into the sampling bottle. Mechanical sludge samplers have been devised and may be used in taking the sample.

Care should be taken in handling and opening sealed bottles containing sludge, especially if it has been undergoing digestion, as gas may accumulate in them.

Raw sewage and sewage effluents should ordinarily be analyzed within 6 hours after sampling. Polluted water should be tested within 12 hours. Certain determinations, such as that for dissolved oxygen, should be made at once. If the samples are kept at low temperatures, the extent of the changes taking place in them will be lessened materially.

The tests made at a sewage plant depend largely upon the size of the plant. Many small plants do not have extensive laboratories or equipment. Also, these plants may be operated by only one or two men. Due to lack of personnel and equipment many of the desirable tests cannot be made.

The tests that can be made at sewage plants where the operator has little or no chemical training are the following: (1) settleable solids, (2) relative stability or dissolved oxygen determination, (3) pH value, (4) residual chlorine. Since the dissolved oxygen test is more difficult than the relative stability test, the latter is recommended for use at a small plant.

The determination of settleable solids by Imhoff cones indicates the amount of suspended solids in sewage that can be removed by sedimentation. This test also furnishes a quick way of determining the amount of settleable solids removed in sedimentation tanks. Although this test is not an exact laboratory method and gives widely varying results from duplicate determinations, the test has been extensively used.

Sewage will putrefy or become septic due to bacterial action and a lack of oxygen. This susceptibility of a sewage to putrefy is known as putrescibility and the test to indicate this characteristic of sewage is known as the putrescibility test, but more generally referred to as the relative stability test.

This test, which is simple to perform, has considerable value and may be made on effluents that have received complete treatment. The test is not intended to be used on raw or settled sewage. If the sewage contains an appreciable amount of suspended or colloidal matter or germicides such as copper salts, bisulfide, calcium hypochlorite or free chlorine, the test has no value. Furthermore, the test should not be made on lime-precipitated effluents containing free caustic alkalinity. This test is only a relative test. It expresses the ratio in percentage of the oxygen available to the amount required to satisfy the complete biochemical oxidation. This test is being superseded by the more desirable B.O.D. test, but since it is easy to perform and requires a minimum of apparatus it is still used extensively as a guide to the character of a treated or partially treated sewage.

The dissolved oxygen test is used to determine the oxygen in the sewage effluent. If dissolved oxygen is found in sufficient concentration in the effluent from a sewage treatment plant it means that, as long as the oxygen is present, putrefactive odors will not be given off. The test may be influenced because of the possible presence of reducing agents other than carbonaceous organic matter. The test has a limited value for quick information as an aid in the control of treatment-plant operation where the tests are made under similar and controllable conditions. This test will also give significant data when made in connection with the B.O.D. and relative stability tests.

Determining the pH value of raw and digested sludge is of value in the control of digestion tanks. This test, when made on raw sewage, will sometimes indicate whether or not industrial wastes containing acids or alkalies are being discharged into the sewerage system.

In case sewage or sewage effluents are treated with chlorine, it is desirable to determine the amount of residual chlorine present. This test should be made sufficiently often to assure the operator that the proper residual is being maintained. The presence of a small residual of chlorine after a contact period of 10 or 15 minutes is an indication that 99% or more of the bacteria have been destroyed.

Every sewage treatment plant using or contemplating the use of chemical precipitation should have available in its laboratory a stirring apparatus for making jar tests. This test enables the study in the laboratory of dosages and methods of chemical precipitation which can be used as a guide in plant operation. The jar tests should be made each time there is an appreciable change in the quality of the sewage and should be made occassionally as a check even though there is no apparent characteristic change in the sewage. The test is of little value if the quantity of sewage flowing is unknown. The purpose of the test is to determine the minimum dosage giving the best floc and settling results. The chemical feeders must then be set accordingly.

A hand operated stirring device may be purchased from a laboratory supply company for a cost of approximately $50.00. A powered device may be acquired at a cost varying from $120.00 to $200.00. One may be devised by arranging six stirring paddles on a frame. The paddles are turned by a continuous belt powered by a small electric motor. The apparatus should be designed so that the speed may be varied or held constant between 10 and 100 R.P.M.

For a sewage plant, similar to the one in operation at Stillwater, it is recommended that at least the tests mentioned above be performed. If future conditions warrant, it will be desirable for other tests to be included.

The equipment necessary and the present cost to equip a small laboratory is shown in Table I. This table lists the minimum amount of equipment for each of the suggested tests. The number indicates the number of pieces of equipment used in the various tests.

TABLE I

Equipment	Tests: Settleable Solids	Relative Stability	Dissolved Oxygen	pH Value	Unit Cost
Imhoff Cones	3				$ 4.00
Imhoff Cone Stand	1				1.50
Bottles, 8 oz., (glass stoppered)		12	2		.30
Pipettes		2	2		.28
Erlenmeyer Flask (500 ml.)			1		.25
Burette (50 ml.)			1		1.60
pH Comparator and discs				1	20.00
Nessler Tube					.60
Burette Support			1		3.75
Total Cost	$13.50	$4.16	$6.77	$20.00	

Equipment	Residual Chlorine	Jar Test	General	Unit Cost
Erlenmeyer Flask (500 ml.)	1			$ .25
Burette (50 ml.)	1			1.60
Nessler Tube	2			.60
Beakers (2 liters)		6		.85
Beakers (400 ml.)			2	.32
Stirring Device		1		50.00
Thermometers			2	3.00
Funnels (short stem)			2	.24
Funnels (long stem)			2	.48
Ring Stand			1	.90
Rings with Clamp			2	.21
Total Cost	$3.05	$55.10	$9.40	

In plants with better equipped laboratories, and with one or more men trained in the chemistry of sewage, a more extensive examination of the sewage and the sludge is desirable.

Usually the two most important tests are the B.O.D. determination and the determination of the amount of suspended solids present.

Of these two, the biochemical oxygen demand test is perhaps the most widely used in sewage analysis. It is used as a measure of the strength of the sewage and the reduction of the demand is frequently used to determine the plant efficiency. The B.O.D. is a measure of the oxygen required to stabilize the sewage. This test is difficult to make and requires considerable experience if reliable results are to be obtained. However, if accurate results are found, it comes the nearest to giving the true character of the sewage.

It is of value to know the amount of suspended solids. The suspended solids include the settleable solids as well as the non-settleable solids. Because the strength of the sewage is largely dependent upon the amount of organic solids in the sewage, it is desirable to make the determination at various points of the treatment plant to determine the efficiency of the removal units. A well designed sedimentation basin with average detention time of 2 hours or more should normally remove from 50 to 70 per cent of the suspended solids.

The removal of the suspended solids is usually an indication of the adequacy of the physical phase of the sewage-treatment plant, since dissolved solids are not greatly effected by this treating process. Usually the determination of dissolved solids is not made as a routine test, but is made when conditions warrant. It may be necessary to make the test when the sewage contains certain industrial wastes.

Settleable solids, which are often determined by use of Imhoff cones, should be determined by weight to acquire more accurate results. It is also desirable to check the suspended solids in the final effluent to determine its clarity.

The organic nitrogen in sewage represents proteins and intermediate products of decomposition. Some of the total nitrogen in sewage is insoluble and is removed in the sedimentation basin. The total nitrogen is composed of organic nitrogen, ammonia, nitrites and nitrates. Five tests for nitrogen determination that may be made are free ammonia, albuminoid ammonia, total organic nitrogen, nitrites and nitrates. Free ammonia represents the anaerobic bacterial decomposition of organic matter. Usually the raw sewage is high in organic nitrogen, low in ammonia and contains little or no nitrites and nitrates. Since ammonia formation occurs, and the ammonium compounds are soluble in water, there will usually be an increase in ammonia content through the primary sedimentation basin. Nitrites and nitrates are compounds containing oxygen and represent two stages of oxidation. The reduction of ammonia and the formation of nitrates and nitrites begins as oxidation starts to take place. This may take place on the trickling filter, or in the aeration tank of the activated sludge plant.

Nitrites are unstable and are reduced to ammonia or are oxidized into nitrates. Their presence usually indicates that a change is in progress. Nitrates are the most stable compound form of nitrogen and their presence in effluents indicates stability.

Since anearobic action predominates in sludge digestion, there will be little or no nitrites and nitrates.

The various nitrogen tests give an overall picture of plant operation and can be used as a valuable index.

The tests to make and their relative importance will often depend upon the character of the raw sewage and the type of treatment. It is recommended that plants with a well equipped laboratory, make as many of the tests as possible. A record of the tests must be studied and the results that give the best information for the efficient operation of that particular plant, will determine the relative importance of the laboratory analyses.

Sometimes it may be advisable to make microscopic examinations, if the body of water receiving the effluent is used for recreational purposes, or serves as a water supply. However, bacteriological analyses are seldom made in the study of sewage, because it is known that intestinal bacteria are present and that the concentration of bacteria is high. This test would be more valuable to those interested in water supply. Since bacteriological analysis has no significance in sewage plant operation, it would rank least in importance and probably would seldom if ever be made in the sewage plant laboratory.

Table II gives the laboratory equipment used for various tests. Column 1 lists the equipment for a small laboratory, where the minimum recommended tests are made. Column 2 is for an intermediate sized plant with a good laboratory. Column 3 lists the equipment for a larger plant with a well equipped laboratory, where all the recommended tests may be made.

TABLE II

The number indicates the number of pieces of equipment used for the various tests.

Equipment	Laboratory Determinations Column (1) Settleable solids, relative stability, pH, residual chlorine	Column (2) B.O.D. total solids, suspended solids, settleable solids, ash, pH, residual chlorine	Column (3) B.O.D. total solids, suspended solids, settleable solids, ash, pH, residual chlorine, ammonia, nitrate nitrogen, nitrite nitrogen	Unit Cost
Analytical balance	--	1	1	$90.00
Balance weights	--	1	1	17.50
Beakers, 250-ml.	--	--	12	.30
Beakers, 400-ml.	--	6	6	.32
Beakers, 1-liter	--	--	3	.74
Bottles, 250-ml. wide mouth	12	24	48	.60
Bottles, 250-ml. narrow mouth	24	36	36	.60
Bottles, 500-ml. narrow mouth	--	6	6	.80
Bottles, 1,000-ml. narrow mouth	1	2	8	1.25
Bottles, acid, 5 pt.	3	6	6	.55
Burettes, 50-ml.	--	2	2	1.60
Burette support	--	1	1	3.75
Burner, Bunsen	--	2	2	.95
Cork borers, 3/16 to 9/16 in.	--	--	9	1.25

TABLE II
(Continued)

Equipment	Column (1) (Cont.)	Column (2) (Cont.)	Column (3) (Cont.)	Unit Cost
Crucibles, Coors porcelain, 15-ml.	---	12	24	$ .17
Crucibles, Gooch, 25-ml.	---	12	24	.45
Crucible holders, Gooch	---	2	2	.45
Crucible tongs, 8-in. long	---	1	1	.40
Desiccator with plate, 6-in.	---	1	1	15.00
Drying oven	---	1	1	70.00
Evaporating dishes, 85-mm.	---	6	6	.62
Filter paper for Buchner funnel packs	---	---	3	1.50
Funnels, glass, short stem, 2-in.	---	2	4	.24
Funnels, glass, short stem, 5-in.	1	2	2	.48
Funnels, Buchner, 80-mm.	---	---	2	3.10
Glass rods, 6-mm. (ft.)	---	3	3	.90
Glass tubing, 7-mm. (ft.)	---	12	12	.40
Graduated cylinders, 50-ml.	---	2	2	.65
Graduated cylinders, 100-ml.	---	2	2	.75
Graduated cylinders, 250-ml.	---	2	2	1.10

TABLE II
(Continued)

Equipment	Column (1) (Cont.)	Column (2) (Cont.)	Column (3) (Cont.)	Unit Cost
Graduated cylinders, 500-ml.	--	2	2	$ 1.50
Graduated cylinders, 1-liter	--	2	2	2.20
Flasks, Erlenmeyer, 500-ml.	--	2	2	.25
Flasks, flat-bottomed 1-liter	--	--	2	.34
Flasks, volumetric, 250-ml.	--	--	1	.60
Flasks, volumetric, 500-ml.	--	1	1	.70
Flasks, volumetric, 1-liter	--	--	1	1.00
Hot plate, electric, 9 - x 12-in.	--	--	1	3.00
Hydrogen-ion apparatus	1	1	1	17.50
Imhoff cones, 1-liter	3	4	6	3.50
Incubator, 20° C	1	1	1	240.00
Indicator dropping bottles, 50-ml.	--	--	2	.35
Nessler tubes, 50-ml.	--	--	12	.60
Nessler-tube stand	--	--	1	3.50
Orthotolidin testing set	1	1	1	20.00
Pipettes, 2-ml.	--	2	3	.28
Pipettes, 5-ml.	--	2	3	.28

TABLE II
(Continued)

Equipment	Column (1) (Cont.)	Column (2) (Cont.)	Column (3) (Cont.)	Unit Cost
Pipettes, 10-ml.	--	2	3	$ .35
Pipettes, graduated, 1/10, 1-ml.	2	2	6	.45
Pipettes, graduated, 1/100, 1-ml.	--	--	2	.45
Pipettes, graduated, 1/10, 5-ml.	--	2	2	.45
Rings with screw clamp, 3-in.	1	2	2	.21
Ring stand	1	1	2	.90
Rubber tubing, heavy wall, 5-mm. (ft.)	--	6	6	.15
Rubber stoppers for suction flasks	--	2	4	.03
Spatulas, 3-in. blade	--	1	1	.30
Suction flask, 500-ml.	--	2	2	4.80
Suction hose, (ft.)	--	6	3	.25
Suction pump, water	--	1	1	40.00
Test tubes, 25 - x 200-mm. (each)	--	--	12	.06
Test tubes stand	--	--	1	1.80
Thermometer	--	1	2	3.00
Triangles, 2-in. long	--	2	2	.30
Wash bottles, 1-liter	1	1	1	.70
Watch glasses, 2½-in.	--	2	4	.04
Total Cost	$315.69	$638.05	$708.74	

Each sewage laboratory should be equipped with a reference library. This may vary from a few volumes in a small plant to a number in a large one.

A manual giving the step-by-step procedure of all the tests made in sewage treatment should be available. A few recently published books concerning water and sewage should be on hand for reference. It is also desirable if current publications are available. This enables the operators to follow new developments in sewage plant operation.

The following list includes the recommended manuals, books and magazines:

Laboratory Manuals: Analysis of Water and Sewage by Theroux Eldridge and Mallmann. Standard Methods for the Examination of Water and Sewage by American Public Health Association.

Reference Books: Sewage Treatment by Imhoff and Fair. Sewerage and Sewage Treatment by Babbitt.

Magazines: Sewage Works Journal, Official Publication of the Federation of Sewage Works Association. Sewage Works Engineering, Publication by Case, Shepperd and Mann. Water and Sewage Works, Publication by Gillette Publishing Company. Public Works, Publication by Public Works Journal Corp.

BIBLIOGRAPHY

Books

Babbitt, Harold E. Sewerage and Sewage Treatment. New York, N. Y.: John Wiley and Sons, Inc., 1947.

Buswell, Arthur M. The Chemistry of Water and Sewage Treatment. New York, N. Y.: The Chemical Catalog Company, Inc., 1928.

Fuller, George W. and McClintock, James R. Solving Sewage Problems. York, Pa.: McGraw-Hill Book Co., 1926.

Imhoff, Karl and Fair, Gordon Maskew. Sewage Treatment. New York, N. Y.: John Wiley and Sons, Inc., 1940.

Keefer, C. E. Sewage-Treatment Works. New York, N. Y.: McGraw-Hill Book Co., 1940.

Metcalf, Leonard and Eddy, Harrison P. Sewerage and Sewage Treatment. New York, N. Y.: McGraw-Hill Book Company, 1935.

Texas Water Works and Sewerage Short School. Manual for Sewage Plant Operators. Lancaster, Pa.: Lancaster Press, Inc., 1946.

Theroux, Frank R. and Eldridge, Edward F. and Mallmann, W. LeRoy. Analysis of Water and Sewage. New York, N. Y.: McGraw-Hill Book Company, Inc., 1943.

Catalogs

Laboratory Equipment Catalog. Cleveland, Ohio: The Chemical Rubber Co., 1949.

General Catalog of Laboratory Apparatus and Scientific Instruments. Chicago, Ill.: Central Scientific Company.

Welch Catalog of Scientific Apparatus Chemicals and Supplies. Chicago, Ill.: W. M. Welch Scientific Company, 1949.